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10/088969		PCT/EP00/09128		47192/270460	
17. <input checked="" type="checkbox"/> The following fees are submitted				CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):					
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO				\$1,040.00	
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO				\$890.00	
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International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)				\$710.00	
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CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	15	00	X \$18.00	\$.00	
Independent claims	01	00	X \$84.00	\$.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$280.00	\$.00	
TOTAL OF ABOVE CALCULATIONS =				\$1,020.00	
Reduction of 1/2 for filing by small entity, if applicable.				--	\$.00
SUBTOTAL =				\$1,020.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$.00	
TOTAL NATIONAL FEE =				\$1,020.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40 per property				\$.00	
TOTAL FEES ENCLOSED =				\$1,020.00	
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<p>a. <input checked="" type="checkbox"/> Check in the amount of \$1,020.00 is enclosed.</p> <p>b. <input type="checkbox"/> Please charge my Deposit Account No. 11-0855 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 11-0855.</p>					
<p>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p>					
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IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

Applicants: Matthias KATTER and Wilhelm FERNENGEL

International
Application No.: PCT/EP00/09128

U.S. Serial No.:

International
Filing Date: 18 September (18.09.00)

U.S. Filing Date: 25 March 2002 (25.03.02)

For: BORON-LOW Nd-Fe-B ALLOY AND METHOD FOR PRODUCING
PERMANENT MAGNETS ON THE BASIS OF SAID ALLOY

Box PCT
Commissioner for Patents
Washington, D.C. 20231

Attorney Docket No. 47192/270460
Date: 25 March 2002

PRELIMINARY AMENDMENT

Sir:

In the Specification

Kindly amend the above-identified patent application prior to examination.

On page 1, line 2, immediately following the heading "Description" kindly insert the
following paragraph:

--This application claims priority to German Application No. 199 45 942.8 filed on
September 24, 1999 and International Application No. PCT/EP00/009128 filed on September 18,
2000.--

In the Claims

Kindly rewrite the claims as follows:

Express Mail Label No. EL209599976US
 U.S. National Phase Entry of PCT/EP00/09128
 "Boron-Low Nd-Fe-B Alloy and Method for Producing Permanent
 Magnets on the Basis of Said Alloy"
 Filed: 25 March 2002
 PRELIMINARY AMENDMENT

1. (Amended) An alloy comprising at least one rare earth, including yttrium, made of iron, B, Co, Cu, Ga, and Al, as well as of production-based contaminations, whereby the following relations apply to the effective rare earth content $[SE]_{eff}$, the effective boron content $[B]_{eff}$, the mutual content of Dy, Tb, and Ho $[Dy + Tb + Ho]$, the cobalt content $[Co]$, the copper content $[Cu]$, the gallium content $[Ga]$, and the aluminum content $[Al]$:

$$\begin{aligned} 26.9 \text{ wt.}\% &\leq [SE]_{eff} \leq 33 \text{ wt.}\% \\ 2.185 - 0.0442 [SE]_{eff} &\leq [B]_{eff} \leq 1.363 - 0.0136 [SE]_{eff} \\ [Dy + Tb + Ho] &\leq 17 \text{ wt.}\% \\ 0.5 \text{ wt.}\% &\leq [Co] \leq 5 \text{ wt.}\% \\ 0.05 \text{ wt.}\% &\leq [Cu] \leq 0.3 \text{ wt.}\% \\ 0.05 \text{ wt.}\% &\leq [Ga] \leq 0.35 \text{ wt.}\% \\ 0.02 \text{ wt.}\% &\leq [Al] \leq 0.3 \text{ wt.}\% \end{aligned}$$

2. (Amended) The alloy according to claim 1, wherein the following relation applies to the effective boron content $[B]_{eff}$:

$$1.814 - 0.0303 [SE]_{eff} \leq [B]_{eff} \leq 1.363 - 0.0136 [SE]_{eff}$$

3. (Amended) The alloy according to claim 1, wherein the rare earth content $[SE]_{eff}$ is above about 28.9 wt.-%, whereby the following relation applies to the effective boron content:

$$1.814 - 0.0303 [SE]_{eff} \leq [B]_{eff} \leq 1.396 - 0.01491 [SE]_{eff}$$

4. (Amended) The alloy according to claim 1, wherein the rare earth content $[SE]_{eff}$ is above about 28.5 wt.-%, whereby the following relation applies to the effective boron content:

$$1.814 - 0.0303 [SE]_{eff} \leq [B]_{eff} \leq 1.478 - 0.01801 [SE]_{eff}$$

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5. (Amended) The alloy according to claim 1, wherein the rare earth content $[SE]_{eff}$ is above about 28.7 wt.-%.
6. (Amended) The alloy according to claim 1, wherein the alloy has a Co-content of between about 2.5 and about 3.5 wt.-%.
7. (Amended) The alloy according to claim 1, wherein the Cu-content is between about 0.1 and about 0.2 wt.-%.
8. (Amended) The alloy according to claim 1, wherein the Ga-content is between about 0.20 and about 0.30 wt.-%.
9. (Amended) The alloy according to claim 1, wherein the rare earths are selected from a group consisting of the elements Nd, Pr, Dy, and Tb.
10. (Amended) A method for producing permanent magnets from an alloy according to claim 1, comprising:
 - orientation in the magnetic field and pressing of powder that was produced by reduction of at least one melting body, into a blank;
 - sintering of the blank at temperatures between about 1020°C and about 1140°C;

- cooling of the blank to temperatures below about 300°C, whereby cooling occurs at above about 800°C at a median cooling speed $\Delta T_1/\Delta t_1$ of < 5 K/min; and
- starting and cooling of the blank, whereby the following relation applies to the starting temperature T_A in dependency of a median cooling speed $\Delta T_2/\Delta t_2$:
for $\Delta T_2/\Delta t_2 < 5$ K/min:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} < 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

$$460^{\circ}\text{C} \leq T_A \leq 510^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} > 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

for $5 \text{ K/min} \leq \Delta T_2/\Delta t_2 \leq 100 \text{ K/min}$:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C}.$$

11. (Amended) The method according to claim 10, wherein after the sintering process, the blank is maintained at a holding temperature between about 700 and about 800°C for a period of between about half an hour and 2 hours.
12. (Amended) The method according to claim 11, wherein the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of > 5 K/min after the sintering process at holding temperature.
13. (Amended) The method according to claim 12, wherein the cooling speeds $\Delta T_2/\Delta t_2$ and $\Delta T_3/\Delta t_3$ are between about 30 and about 50 K/min.

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14. (Amended) The method according to claim 10, wherein the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of < 5 K/min after the sintering process at holding temperature.

15. (Amended) The method according to claim 14, wherein the cooling speeds $\Delta T_1/\Delta t_1$ to $\Delta T_3/\Delta t_3$ are between about 1 and about 2 K/min.

1. (Amended) An [A] alloy [consisting of] comprising at least one rare earth, including yttrium, made of iron, [consisting of the elements] B, Co, Cu, Ga, and Al, as well as of production-based contaminations, whereby the following relations apply to the effective rare earth content $[SE]_{\text{eff}}$, the effective boron content $[B]_{\text{eff}}$, the mutual content of Dy, Tb, and Ho $[Dy + Tb + Ho]$, the cobalt content $[Co]$, the copper content $[Cu]$, [and] the gallium content $[Ga]$, [as well as] and the aluminum content $[Al]$:

$$\begin{aligned} 26.9 \text{ wt.}\% &\leq [\text{SE}]_{\text{eff}} \leq 33 \text{ wt.}\% \\ 2.185 - 0.0442 [\text{SE}]_{\text{eff}} &\leq [\text{B}]_{\text{eff}} \leq 1.363 - 0.0136 [\text{SE}]_{\text{eff}} \\ [\text{Dy} + \text{Tb} + \text{Ho}] &\leq 17 \text{ wt.}\% \\ 0.5 \text{ wt.}\% &\leq [\text{Co}] \leq 5 \text{ wt.}\% \\ 0.05 \text{ wt.}\% &\leq [\text{Cu}] \leq 0.3 \text{ wt.}\% \\ 0.05 \text{ wt.}\% &\leq [\text{Ga}] \leq 0.35 \text{ wt.}\% \\ 0.02 \text{ wt.}\% &\leq [\text{Al}] \leq 0.3 \text{ wt.}\% \end{aligned}$$

2. (Amended) The [A]alloy according to claim 1, [characterized in that] wherein the following relation applies to the effective boron content [B]_{eff}:

$$1.814 - 0.0303 [\text{SE}]_{\text{eff}} \leq [\text{B}]_{\text{eff}} \leq 1.363 - 0.0136 [\text{SE}]_{\text{eff}}.$$

3. (Amended) The [A]alloy according to claim[s] 1 [or 2], [characterized in that] wherein the rare earth content [SE]_{eff} is above about 28.9 wt.-%, whereby the following relation applies to the effective boron content:

$$1.814 - 0.0303 [\text{SE}]_{\text{eff}} \leq [\text{B}]_{\text{eff}} \leq 1.396 - 0.01491 [\text{SE}]_{\text{eff}}.$$

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4. (Amended) The [A]alloy according to claim[s] 1 [or 2], [characterized in that] wherein
the rare earth content [SE]_{eff} is above about 28.5 wt.-%, whereby the following relation applies to
the effective boron content:

$$1.814 - 0.0303 [\text{SE}]_{\text{eff}} \leq [\text{B}]_{\text{eff}} \leq 1.478 - 0.01801 [\text{SE}]_{\text{eff}}$$

5. (Amended) The [A]alloy according to claim [4] 1, [characterized in that] wherein
the rare earth content [SE]_{eff} is above about 28.7 wt.-%.

6. (Amended) The [A]alloy according to [one of the] claim[s] 1 [to 5], [characterized in
that] wherein the alloy has a Co-content of between about 2.5 and about 3.5 wt.-%.

7. (Amended) The [A]alloy according to [one of the] claim[s] 1 [to 6], [characterized in
that] wherein the Cu-content is between about 0.1 and about 0.2 wt.-%.

8. (Amended) The [A]alloy according to [one of the] claim[s] 1 [to 7], [characterized in
that] wherein the Ga-content is between about 0.20 and about 0.30 wt.-%.

9. (Amended) The [A]alloy according to [one of the] claim[s] 1 [to 8], [characterized in
that] wherein the rare earths are selected from a group consisting of the elements Nd, Pr, Dy, and
Tb.

10. (Amended) A [M]method for producing permanent magnets from an alloy according to [one of the] claim[s] 1, [to 9] [with the following process steps] comprising:

- orientation in the magnetic field and pressing of powder that was produced by reduction of at least one melting body, into a blank;
- sintering of the blank at temperatures between about 1020°C and about 1140°C;
- cooling of the blank to temperatures below about 300°C, whereby cooling occurs at above about 800°C at a median cooling speed $\Delta T_1/\Delta t_1$ of < 5 K/min; and
- starting and cooling of the blank, whereby the following relation applies to the starting temperature T_A in dependency of a median cooling speed $\Delta T_2/\Delta t_2$:

for $\Delta T_2/\Delta t_2 < 5 \text{ K/min}$:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} < 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

$$460^{\circ}\text{C} \leq T_A \leq 510^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} > 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

for $5 \text{ K/min} \leq \Delta T_2/\Delta t_2 \leq 100 \text{ K/min}$:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C}.$$

11. (Amended) The [M]method according to claim 10, [characterized in that] wherein
after the sintering process, the blank is maintained at a holding temperature between about 700
and about 800°C for a period of between about half an hour and 2 hours.

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12. (Amended) The [M]method according to claim 11, [characterized in that] wherein the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of > 5 K/min after the sintering process at holding temperature.

13. (Amended) The [M]method according to claim 12, [characterized in that] wherein the cooling speeds $\Delta T_2/\Delta t_2$ and $\Delta T_3/\Delta t_3$ are between about 30 and about 50 K/min.

14. (Amended) The [M]method according to claim 10 [or 11], [characterized in that] wherein the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of < 5 K/min after the sintering process at holding temperature.

15. (Amended) The [M]method according to claim 14, [characterized in that] wherein the cooling speeds $\Delta T_1/\Delta t_1$ to $\Delta T_3/\Delta t_3$ are between about 1 and about 2 K/min.

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Remarks

Applicants submit that none of the above amendments are being made for reasons related to patentability, but are instead being made to put the application in proper format. No new matter has been added. Applicants also point out that the certain elements indicated in brackets, e.g., [B], [Cu], are not intended to be deleted; this is how they were presented in the original application.

Respectfully submitted



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Description

Boron-low Nd-Fe-B alloy and method for producing permanent magnets on the basis of said alloy.

The invention relates to an alloy on the basis of at least one rare earth, of at least one transition metal and boron, as well as to a method for producing permanent magnets on the basis of said alloy.

Such alloys and methods for producing permanent magnets on the basis of this alloy are known from EP-A-0 124 655. In the known method, an alloy on the basis of neodymium, iron, and boron is initially melted. The alloy is then melted to a melting block, which is then reduced to powder. Blanks are pressed from this powder in a magnetic field, which are then sintered.

For many applications of Nd-Fe-B permanent magnets, especially in motors and all sorts of drives, the coercivity H_{CJ} of 150°C is key to the quality of the permanent magnet. At a low counter field load, the coercivity H_{CJ} at 150°C must be at least 4.5 kOe, better yet more than 5 kOe. At a high counter field load, values above 13 kOe at 150°C are even required. In addition to the high coercivity H_{CJ} , such magnets should also possess a remanence B_r that is as high as possible. For example, the remanence B_r of Nd-Fe-B permanent magnets that have a coercivity H_{CJ} within the area of 4.5 kOe, should be at least 1.29 T at ambient temperature, but better yet more than 1.35 T.

Applications in motors demand the additional requirement that the reversible temperature coefficient of the remanence TK (B_r) must be better than -0.11 %/K within a temperature range of 20°C to 150°C. Also, such permanent magnets should possess a corrosion

resistance that is as good as possible, in order to eliminate complicated and expensive coatings. It is required, for instance, that the loss of mass of uncoated magnets in a so-called HAST test should be lower than 1 mg/cm² after ten days. In the HAST test, permanent magnets are exposed to a pressure of 39.16 psi at an ambient temperature of 130°C and a relative humidity of 95%.

These requirements are currently not being met by conventional Nd-Fe-B permanent magnets.

Based on this prior art, the invention is based on the task of creating an alloy for permanent magnets on the basis of at least one rare earth, and of at least one transition metal and boron that has a higher coercivity H_{CJ} than conventional alloys at the same remanence B_r , and possesses a lower temperature coefficient of the remanence, and is corrosion resistant.

This task is solved by the invention by the use of an alloy possessing the characteristics stated in claim 1.

Conventional Nd-Fe-B alloys essentially consist of three phases: the magnetically hard ϕ -phase with the compound $Nd_2Fe_{14}B$, the nonmagnetic η -phase with the compound $Nd_{1.1}Fe_4B_4$, and the nonmagnetic filler phase, which almost exclusively consists of Nd. The Nd-rich filler phase separates the particles of the ϕ -phase from each other magnetically, which results in a high coercivity H_{CJ} . However, in a B concentration that is too low causes the risk of formation of a magnetically soft Nd_2Fe_{17} phase instead of the nonmagnetic η -phase, which substantially reduces the coercivity H_{CJ} . Unlike in conventional Nd-Fe-B alloys, the alloys produced according to the invention, the Nd_2Fe_{17} phase harmful for the coercivity H_{CJ} is not created instead of the nonmagnetic η -phase when a critical B-content is not reached, but instead initially a series of

nonmagnetic phases containing Ga. Contrary to the ferromagnetic $\text{Nd}_2\text{Fe}_{17}$ phase, these phases containing Ga contribute to the magnetic isolation of the particles of the ϕ -phase, thus improving the coercivity H_{CJ} and also the temperature dependency of the alloy.

The invention is further based on the task of stating a method for producing permanent magnets on the basis of said alloy.

This task is solved by the invention by means of a method with the characteristics listed in claim 6.

Particularly high values can be achieved for the coercivity H_{CJ} with a skillfully adjusted temperature. It should be highlighted that particularly well values can be achieved for the coercivity H_{CJ} especially with rapid cooling. Rapid cooling, however, goes hand in hand with an effective use of the ovens. Therefore, even large permanent magnet components can be produced with slow cooling, without the formation of cooling tears in the permanent magnet components, and without a substantial decrease of the coercivity H_{CJ} .

The invention is explained in detail in the following attached drawings. They show:

- Figure 1 a section from a phase diagram for Nd-Fe-B permanent magnets;
- Figure 2 an illustration of the correlation between remanence B_r and coercivity H_{CJ} for various Nd-Fe-B permanent magnets;
- Figure 3 a diagram with a temperature adjustment during the sintering and starting procedures;

- Figure 4 an additional diagram with an additional possible temperature adjustment during the sintering and starting procedures;
- Figure 5 an illustration showing the dependency of the coercivity H_{CJ} from the type of temperature adjustment during the sintering and starting procedures;
- Figure 6 a diagram showing the dependency of the remanence B_r from the effective contents of boron and rare earths;
- Figure 7 an illustration showing the dependency of the coercivity H_{CJ} at 150°C of the effective content of boron and rare earths during slow cooling;
- Figure 8 an illustration showing the dependency of the temperature coefficient of the coercivity TK (H_{CJ}) from the effective content of boron and rare earths during slow cooling;
- Figure 9 an illustration showing the dependency of the coercivity H_{CJ} at 150°C of the effective content of boron and rare earths during rapid cooling; and
- Figure 10 an illustration showing the dependency of the temperature coefficient TK (H_{CJ}) of the coercivity H_{CJ} of the effective content of boron and rare earths with rapid cooling.

Figure 1 is a phase diagram showing the composition of an Nd-Fe-B alloy in dependency of the effective content of boron and rare earths. The structure suitable for the application as a permanent magnet particularly exists within a phase triangle 1. The alloy consists of magnetically hard particles of the ϕ -phase with the composition $Nd_2Fe_{14}B$ within this phase triangle 1, as well as of particles of the nonmagnetic η -phase with the composition

$\text{Nd}_{1.1}\text{Fe}_4\text{B}_4$, and the nonmagnetic filler phase, which almost exclusively consists of Nd. The Nd-rich filler phase separates the particles of the ϕ -phase from each other magnetically, which is necessary in achieving a high coercivity H_{CJ} .

In order to evaluate whether a certain compound of the alloy exists inside or outside of the phase triangle 1, it is initially necessary to correct the content of rare earths and boron with regard to contaminations, as a part of the Nd is bound in the form of Nd-oxides, Nd-nickel-carbides, and Nd-nitrides. The effective content of rare earth $[\text{SE}]_{\text{eff}}$, and the effective content of boron $[\text{B}]_{\text{eff}}$ is derived from the following formulas:

$$[\text{SE}]_{\text{eff}} = ([\text{SE}] - [\Delta \text{SE}]) f,$$

$$[\text{B}]_{\text{eff}} = [\text{B}] f,$$

whereby $[\text{SE}]$ and $[\text{B}]$ each are the weight parts of rare earth and boron. $[\Delta \text{SE}]$ is therefore the part of rare earth that is bound in the compounds Nd_2O_3 , Nd_2CO , and NdN . f is a standardization factor.

$$[\Delta \text{SE}] = 5.993 [\text{O}] + 16.05 [\text{C}] + 10.30 [\text{N}]$$

$$f = 100 / ([100 - [\Delta \text{SE}] - [\text{O}] - [\text{C}] - [\text{N}]]).$$

$[\text{O}]$, $[\text{C}]$, and $[\text{N}]$ are the weight parts of O, C, and N. All concentration values in the formulas above are in weight-%.

The effective content of rare earth and boron influences the composition of the structure. In point η of the phase triangle 1, the structure exists almost exclusively in the form of the η -phase. In point ϕ of the phase triangle 1, the alloy is in the ϕ -phase, while in point SE it essentially consists of the Nd-rich filler phase. Principally, the part of the η -phase can be of any low value. With a boron content that is too low, however, there is a risk that

The magnetically soft $\text{Nd}_2\text{Fe}_{17}$ -phase forms instead of the nonmagnetic η -phase, which substantially reduces the coercivity H_{CJ} . The compound of the Nd-Fe-B permanent magnets has therefore traditionally always been chosen so that it lies within the phase triangle 1, especially above the tie-line 2. The values for each point in the phase diagram in figure 1 are listed in table 1.

	SE in wt.-%	B-content in wt.-%	Fe-content in wt.-% residue
	SE in Gew.%	B-Gehalt in Gew.%	Fe-Gehalt in Gew.%
φ	26,68	1,000	Rest
η	37,3	10,2	Rest
Nd-reich	98	0	Rest
$\text{Nd}_2\text{Fe}_{17}$	23,3	0	Rest

Table 1

For many applications of Nd-Fe-B permanent magnets, however, and especially in motors and all sorts of drives, the coercivity H_{CJ} is essential. The coercivity H_{CJ} of the Nd-Fe-B permanent magnet used should be at least 4.5 kOe, better yet at least 5 kOe with low counter field load. Higher values above 13 kOe at 150°C are required with higher counter field loads. In addition to a high coercivity H_{CJ} at a temperature of 150°C, such Nd-Fe-B permanent magnets should also have a remanence B_r that is as high as possible.

Particularly for the application in motors it is required that the reversible temperature coefficient of the remanence TK (B_r) within the temperature range of 20°C to 150°C should be better than -0.11 %/K.

Additionally, Nd-Fe-B permanent magnets should have a corrosion resistance that is as well as possible, in order to eliminate complicated and expensive coating.

It was discovered that a phase area 3 forms by adding gallium to the alloy below the tie-line 2, by the fact that in addition to the magnetically hard ϕ -phase, and in addition to the nonmagnetic, Nd-rich phase, further phases containing Ga exist. A tie-line 4 separates the phase area 3 from another phase area 5, because the $\text{Nd}_2\text{Fe}_{17}$ -phase is predominant. Surprisingly, it is now possible to meet the requirements of the use of Nd-Fe-B permanent magnets in motors with the alloys in the phase area 3. This improvement can be explained by the following metallurgic model: In conventional Nd-Fe-B permanent magnets, the magnetically soft $\text{Nd}_2\text{Fe}_{17}$ -phase that is harmful for the coercivity H_{CJ} , if the critical boron content falls short, as illustrated by the border line 2. When adding gallium, cobalt, and copper to the Nd-Fe-B alloy, not the $\text{Nd}_2\text{Fe}_{17}$ -phase is created instead of the nonmagnetic η -phase with the values falling short of the border line 2, but initially a series of nonmagnetic phases containing Ga are created. Contrary to the $\text{Nd}_2\text{Fe}_{17}$ -phase, these phases containing Ga contribute to the magnetic isolation of the particles of the ϕ -phase. This improves the coercivity H_{CJ} , and also its temperature coefficient. A further reduction of the boron contents finally leads to the formation of the $\text{Nd}_2\text{Fe}_{17}$ -phase in the phase area 5, and therefore to the breakdown of the coercivity H_{CJ} .

In addition to gallium, Co and Cu may also be added to the alloy with beneficial effect.

For example, by adding Co to the alloy, the temperature coefficient of the remanence TK (B_r) of Nd-Fe-B permanent magnets can be improved. In particular the temperature coefficient of the remanence TK (B_r) can be improved from $-0.12\text{ \%}/\text{K}$ to approximately $-0.105\text{ \%}/\text{K}$ by adding 3 wt.-% of Co to the alloy. However, if only Co is added to the alloy, this leads to the formation of a magnetically soft SECo_2 -Laves-phase, thereby

drastically reducing the coercivity H_{CJ} . The formation of this harmful Laves-phase can be prevented by adding Cu at the same time. It has been found that the addition of 0.05 to 0.2 wt.-% of Cu is beneficial. Furthermore, Nd-Fe-B permanent magnets containing Cu can be slowly cooled after a heating treatment performed during the production process, without essentially reducing the coercivity H_{CJ} .

The resistance of Nd-Fe-B permanent magnets to corrosion by water vapor is improved by about three orders of magnitude by additionally adding Co, Cu, and Ga to the alloy as opposed to conventional Nd-Fe-B permanent magnets. In this process, a particularly reactive Nd-rich filler phase is largely replaced by chemically more noble phases containing Co, Cu, and Ga.

These measures result in Nd-Fe B permanent magnets showing a loss of mass of $< 1 \text{ mg/cm}^2$ based on the surface of the Nd-Fe-B permanent magnet after ten days in a so-called HAST test. In the so-called HAST test, the Nd-Fe-B permanent magnets are exposed to a pressure of 39.16 psi at a temperature of 130°C and a relative humidity of 95%.

It is also possible to increase the coercivity H_{CJ} by replacing one part of the Nd with Dy, Tb, or Ho, without essentially changing the content ratio of rare earth to the Fe and B content. As, contrary to Nd, the magnetic moment of Dy, Tb, and Ho is oriented anti-parallel to the magnetic moment of Fe, this inevitably leads to a reduction of the achievable remanence B_r . This means that the increase of the coercivity H_{CJ} is tied to the decrease of the remanence B_r .

This connection is illustrated in figure 2 and the respective table 2.

Alloy

Legierung	SE, effektiv	B, effektiv	Dy	Co	Cu	Ga	H _{CJ} (20°C, kOe)	H _{CJ} (150°C, kOe)	B _r (20°C, T)
A 1	29,2	0,98	3	-	-	-	17	3,5	1,33
A 2	29,5	0,98	4,6	-	-	-	21	5	1,28
A 3	29,6	0,98	6,5	-	-	-	26	8	1,22
A 4	29,7	0,98	8,6	-	-	-	31	11	1,16
B 1	29,3	0,94	3	3	0,15	0,23	18	5,3	1,35
B 2	29,5	0,94	5,5	3	0,15	0,23	23	8	1,28
B 3	30	0,93	9,5	3	0,15	0,23	31	13	1,18

Table 2

The alloys A1 to A4 represent conventional alloys with compounds as listed in table 2. Alloys B1 to B3 are alloys according to the invention. Figure 2 clearly shows that although the coercivity increases with an increased content of Dy, the remanence decreases.

Furthermore, figure 2 shows that those alloys to which Co, Cu and Ga were added, have a higher coercivity H_{CJ} at the same amount of remanence B_r in comparison to conventional alloys. The latter does not apply to ambient temperature, but instead especially to a temperature of 150°C.

Nd-Fe-B alloys with a Dy content of around 3 wt.-% have now been examined systematically. The results of these tests are listed in tables 3 and 4. It was discovered during the course of these tests that the magnetic characteristics of Nd-Fe-B permanent magnets largely depend on the temperature adjustment during the course of the heat treatment of the manufacturing process.

Nd-Fe-B alloys are usually produced by initially melting the alloy together with the desired compounds, and then decanted to a melting block. The melting block is then

The high amounts of coercivity H_{CJ} , which result despite of slow cooling at a low effective content of boron of 0.92 wt.-%, are of particular interest. This is beneficial especially when the Nd-Fe-B permanent magnets containing large sectional surfaces are to be produced, because only low cooling speeds $\Delta T/\Delta t$ of < 10 K/min are permissible for such components during the sintering process and the heat treatment in order to avoid cooling rips. These low cooling speeds, however, may lead to slight imperfections of the magnetic characteristics only. According to figure 5 it is quite possible to slowly cool

down Nd-Fe-B permanent magnets at cooling speeds within a range of 1 to 2 K/min after the heat treatment, without sustaining substantial imperfections of the magnetic characteristics, as long as the Nd-Fe-B alloy is low in boron. An Nd-Fe-B alloy low in boron is an alloy with an effective boron content of below the tie-line 2.

Tables 3 and 4 contain a list of compounds and magnetic characteristics of isostatically pressed Nd-Fe-B permanent magnets with various effective contents of rare earths and boron. The statements in bold font refer to the alloys low in boron content according to the invention. All Nd-Fe-B permanent magnets have been produced according to the common powder-metallurgic process, and were sintered to a density of $> 7.6 \text{ g/cm}^3$ at a temperature of approximately 1060°C . The Nd-Fe-B permanent magnets listed in table 3 were slowly cooled to ambient temperature at approximately 1 to 2 K/min. They were then tempered at a temperature of 440°C to 560°C for one to two hours, and again slowly cooled to ambient temperature at approximately 1 to 2 K/min. The magnets listed in table 4 were initially quenched slowly from the sintering temperature to approximately 750°C at about 2 K/min, and after a waiting period of about 1 hour, they were rapidly quenched to ambient temperature at approximately 30 to 50 K/min. These Nd-Fe-B permanent magnets were then again rapidly cooled to ambient temperature at approximately 30 to 50 K/min after subsequent tempering at 470 to 530°C .

Figure 6 contains the values for the remanence B_r for the alloys in table 3 in dependency of the effective content of boron and rare earths. Two level lines illustrate the tendency of the increasing remanence B_r at decreasing effective rare earth content, and increasing effective boron content. A remanence B_r of over 1.35 T is achieved for isostatically pressed Nd-Fe-B permanent magnets with an effective rare earth content of $< 30 \text{ wt.-%}$, and an effective boron content of $> 0.93 \text{ wt.-%}$. With regard to the boron contents, the

remanence B_r goes through a maximum just below the threshold 2 to the phase triangle 1.

Figure 7 illustrates the dependency of the coercivity at 150°C for the slowly cooled Nd-Fe-B permanent magnets in table 3. Figure 7 proves that the coercivity H_{CJ} increases at 150°C with a decreasing effective boron content. The same applies for the coercivity at 20°C.

Finally, figure 8 shows the dependency of the temperature coefficient of H_{CJ} for slowly cooled Nd-Fe-B permanent magnets in dependency of an effective content of rare earths and boron. Here too, increasingly better values are achieved for the temperature coefficient with decreasing effective boron content. Together with the increasing coercivity H_{CJ} , this leads to an increase of the coercivity H_{CJ} at 150°C of below 4.5 kOe to values of up to above 5.5 kOe for slowly cooled magnets. These particularly high values for the coercivity H_{CJ} are especially beneficially particularly for a rare earth content $[SE]_{\text{eff}}$ of more than 28.9 wt.-%, while the following relation applies for the effective boron content:

$$1.814 - 0.0303 [\text{SE}]_{\text{eff}} \leq [\text{B}]_{\text{eff}} \leq 1.396 - 0.01491 [\text{SE}]_{\text{eff}}$$

The same applies to Nd-Fe-B permanent magnets that are rapidly cooled from approximately 750°C and from starting temperature. However, according to figures 9 and 10, slightly better values are achieved not only for the temperature dependency, but also for the absolute values as compares to the slowly cooled Nd-Fe-B permanent magnets. This results in an expanded area in which the required characteristics, namely a remanence $B_r > 1.35$ T at ambient temperature, and a coercivity $H_{CJ} > 5$ kOe at 150°C are achieved.

Particularly high values for the coercivity H_{CJ} at 150°C are achieved for an effective content of rare earth of above 28.5 wt.%, especially 28.7 wt.%, while the following

relation applies to the effective boron content:

$$1.814 - 0.0303 [\text{SE}]_{\text{eff}} \leq [\text{B}]_{\text{eff}} \leq 1.478 - 0.01801 [\text{SE}]_{\text{eff}}$$

In conclusion it should be noted that Pr may also be used in addition to Nd, without negatively influencing the magnetic characteristics of the permanent magnets.

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Compound in wt.-%

starting
temp.

Zusammensetzung in Gew.-%						Anlaß- temp.	B _r (20°C)	(BH) _{max}	H _{CJ} (20°C)	H _{CJ} (150°C)	TK(H _{CJ}) (20-150°C)
SE, effektiv	B, effektiv	Dy	Co	Cu	Ga	(°C)	(T)	(MGOe)	(kOe)	(kOe)	(%/K)
28,1	0,99	2,8	3,1	0,15	0,22	470	1,386	46,6	16,16	4	-0,579
28,1	0,99	2,8	3,1	0,15	0,22	500	1,372	45,7	15,06		-0,570
28,1	0,99	2,8	3,1	0,15	0,22	530	1,382	46,4	15,57		
28,9	0,98	2,8	3	0,13	0,2	470	1,383	46,4	16,88	4,08	-0,583
28,9	0,98	2,8	3	0,13	0,2	500	1,378	46,1	17,24	4,39	-0,573
28,9	0,98	2,8	3	0,13	0,2	530	1,391	47,0	16,4	3,84	-0,589
29,6	0,97	2,8	2,9	0,1	0,18	470	1,376	46,0	16,27	4,02	-0,579
29,6	0,97	2,8	2,9	0,1	0,18	500	1,36	44,9	16,63	4,11	-0,579
29,6	0,97	2,8	2,9	0,1	0,18	530	1,374	45,8	9,96		
28,7	0,94	2,9	3,1	0,17	0,22	500	1,374	45,8	15,69	4,42	-0,553
28,65	0,95	2,9	3,1	0,16	0,22	500	1,356	44,6	16,43	4,51	-0,558
28,6	0,96	3	3,2	0,16	0,22	500	1,375	45,9	16,89	4,59	-0,560
28,55	0,97	3	3,2	0,15	0,22	500	1,375	45,9	17,58	4,42	-0,576
28,5	0,98	3	3,2	0,15	0,21	500	1,382	46,4	17,15	4,42	-0,571
29,8	0,92	3,1	3,0	0,16	0,22	500	1,341	43,6	18,08	5,36	-0,541
29,8	0,93	3,1	3,0	0,15	0,22	500	1,352	44,4	18,24	5,26	-0,547
29,8	0,95	3,1	3,0	0,15	0,22	500	1,355	44,6	18,11	5,04	-0,555
29,8	0,96	3,1	3,0	0,14	0,22	500	1,363	45,1	17,34	4,49	-0,570
29,8	0,98	3,1	3,0	0,14	0,22	500	1,348	44,1	17,42	4,41	-0,574
29,9	0,94	3,3	3,1	0,14	0,21	440	1,369	45,5	15,95	3,79	-0,586
29,9	0,94	3,3	3,1	0,14	0,21	470	1,342	43,7	17,71	4,67	-0,566
29,9	0,94	3,3	3,1	0,14	0,21	500	1,353	44,4	17,79	4,6	-0,570
29,9	0,94	3,3	3,1	0,14	0,21	530	1,352	44,4	10,62		
29,9	0,94	3,3	3,1	0,14	0,21	560	1,311	41,7	9,55		
29,2	0,93	2,9	3	0,19	0,25	470	1,364	45,2	16,56	4,67	-0,552
29,2	0,93	2,9	3	0,19	0,25	500	1,351	44,3	17	4,95	-0,545
29,2	0,93	2,9	3	0,19	0,25	530	1,366	45,3	16,38	5	-0,534
29,5	0,93	2,9	3	0,17	0,23	470	1,347	44,0	17,57	5,12	-0,545
29,5	0,93	2,9	3	0,17	0,23	500	1,331	43,0	18,21	5,39	-0,542
29,5	0,93	2,9	3	0,17	0,23	530	1,344	43,8	17,97	5,42	-0,537
29,9	0,92	2,9	3	0,16	0,22	470	1,341	43,6	18,62	5,42	-0,545
29,9	0,92	2,9	3	0,16	0,22	500	1,331	43,0	19,08	5,71	-0,539
29,9	0,92	2,9	3	0,16	0,22	530	1,307	41,5	18,56	5,61	-0,537

Table 3

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Compound in wt.-%

starting
temp.

Zusammensetzung in Gew.%						Anlaß- temp.	B _r (20°C)	(BH) _{max}	H _{cJ} (20°C)	H _{cJ} (150°C)	TK(H _{cJ}) (20-150°C)
SE, effektiv	B, effektiv	Dy	Co	Cu	Ga	(°C)	(T)	(MGOe)	(kOe)	(kOe)	(%/K)
28,7	0,94	2,9	3,1	0,17	0,22	500	1,37	45,6	17,16	4,99	-0,546
28,65	0,95	2,9	3,1	0,16	0,22	500	1,341	43,6	18,02	5,15	-0,549
28,6	0,96	3	3,2	0,16	0,22	500	1,374	45,8	17,43	4,9	-0,553
28,55	0,97	3	3,2	0,15	0,22	500	1,372	45,7	16,33	4,61	-0,552
28,5	0,98	3	3,2	0,15	0,21	500	1,362	45,0	16,69	4,79	-0,551
29,8	0,92	3,1	3	0,16	0,22	500	1,343	43,8	18,3	5,59	-0,534
29,8	0,93	3,1	3	0,15	0,22	500	1,351	44,3	18,46	5,5	-0,539
29,8	0,95	3,1	3	0,15	0,22	500	1,35	44,2	18,17	5,18	-0,550
29,8	0,96	3,1	3	0,14	0,22	500	1,354	44,5	16,87	4,71	-0,554
29,8	0,98	3,1	3	0,14	0,22	500	1,344	43,8	16,91	4,78	-0,552
28,8	0,95	3	2,8	0,14	0,26	500	1,359	44,8	18,65	5,66	-0,536
28,8	0,95	3	2,8	0,14	0,26	530	1,361	45,0	18,22	5,67	-0,530
29,2	0,93	2,9	3	0,19	0,25	470	1,354	44,5	18,61	5,65	-0,536
29,2	0,93	2,9	3	0,19	0,25	500	1,343	43,8	18,87	5,67	-0,538
29,2	0,93	2,9	3,0	0,19	0,25	530	1,355	44,6	18,73	5,82	-0,530
29,5	0,93	2,9	3,0	0,17	0,23	470	1,342	43,7	19,71	5,83	-0,542
29,5	0,93	2,9	3,0	0,17	0,23	500	1,323	42,5	19,56	5,92	-0,536
29,5	0,93	2,9	3,0	0,17	0,23	530	1,329	42,9	19,9	6,09	-0,534
29,9	0,92	2,9	3	0,16	0,22	470	1,337	43,4	20,3	6,09	-0,538
29,9	0,92	2,9	3	0,16	0,22	500	1,343	43,8	19,8	5,9	-0,539
29,9	0,92	2,9	3	0,16	0,22	530	1,335	43,3	20	6,09	-0,535

Table 4

the rare earth content $[\text{SE}]_{\text{eff}}$ is above 28.5 wt.-%, whereby the following relation applies to the effective boron content:



10. Method for producing permanent magnets from an alloy according to one of the claims 1 to 9 with the following process steps:
- orientation in the magnetic field and pressing of powder that was produced by reduction of at least one melting body, into a blank;
 - sintering of the blank at temperatures between 1020°C and 1140°C;
 - cooling of the blank to temperatures below 300°C, whereby cooling occurs at above 800°C at a median cooling speed $\Delta T_1/\Delta t_1$ of < 5 K/min; and

- starting and cooling of the blank, whereby the following relation applies to the starting temperature T_A in dependency of a median cooling speed $\Delta T_2/\Delta t_2$:

for $\Delta T_2/\Delta t_2 < 5 \text{ K/min}$:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} < 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

$$460^{\circ}\text{C} \leq T_A \leq 510^{\circ}\text{C} \text{ for } [\text{B}]_{\text{eff}} < 2.993 - 0.069 [\text{SE}]_{\text{eff}}$$

for $5 \text{ K/min} \leq \Delta T_2/\Delta t_2 \leq 100 \text{ K/min}$:

$$450^{\circ}\text{C} \leq T_A \leq 550^{\circ}\text{C}.$$

11. Method according to claim 10,
characterized in that
after the sintering process, the blank is maintained at a holding temperature between 700 and 800°C for a period of between half an hour and 2 hours.
12. Method according to claim 11,
characterized in that
the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of > 5 K/min after the sintering process at holding temperature.
13. Method according to claim 12,
characterized in that
the cooling speeds $\Delta T_2/\Delta t_2$ and $\Delta T_3/\Delta t_3$ are between 30 and 50 K/min.
14. Method according to claim 10 or 11,
characterized in that
the raw body is cooled at a median cooling speed $\Delta T_3/\Delta t_3$ of < 5 K/min after the sintering process at holding temperature.

15. Method according to claim 14,
characterized in that
 the cooling speeds $\Delta T_1/\Delta t_1$ to $\Delta T_3/\Delta t_3$ are between 1 and 2 K/min.

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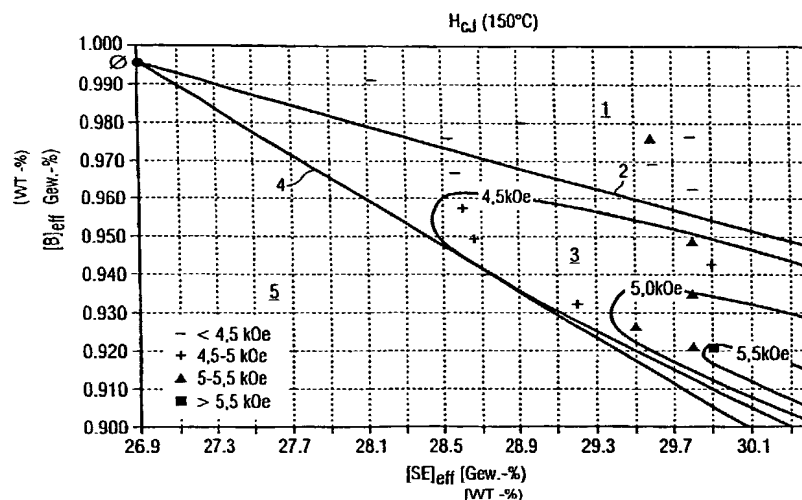
Veröffentlicht:

— Mit internationalem Recherchenbericht.

[Fortsetzung auf der nächsten Seite]

(54) Title: **BORON-LOW Nd-Fe-B ALLOY AND METHOD FOR PRODUCING PERMANENT MAGNETS ON THE BASIS
OF SAID ALLOY**

(54) Bezeichnung: **BORARME Nd-Fe-B-LEGIERUNG UND VERFAHREN ZUR HERSTELLUNG VON DAUER_MAGNETEN
AUS DIESER LEGIERUNG**



(57) Abstract: The invention relates to boron-low Nd-Fe-B permanent magnets that have high coercive forces. To this end, the concentrations have to meet the following requirements: 26,9 wt.-% ≤ [SE]_{eff} ≤ 33 wt.-%; 2,185 - 0,0442 [SE]_{eff} ≤ [B]_{eff} ≤ 1,363 - 1,0136 [SE]_{eff} [Dy + Tb + Ho] ≤ 50 % [SE]_{eff}; 0,5 wt.-% ≤ [Co] ≤ 5 wt.-%; 0,05 wt.-% ≤ [Cu] ≤ 0,3 wt.-%; 0,05 wt.-% ≤ [Ga] ≤ 0,35 wt.-%; 0,02 wt.-% ≤ [Al] ≤ 0,3 wt.-%.

(57) Zusammenfassung: Borarme Nd-Fe-B-Dauermagnete Können hohe Koerzitivfeldstärken aufweisen. Dazu müssen die Konzentrationen folgenden Bedingungen genügen: 26,9 Gew.-% ≤ [SE]_{eff} ≤ 33 Gew.-%; 2,185 - 0,0442 [SE]_{eff} ≤ [B]_{eff} ≤ 1,363 - 1,0136 [SE]_{eff} [Dy + Tb + Ho] ≤ 50 % [SE]_{eff}; 0,5 Gew.-% ≤ [Co] ≤ 5 Gew.-%; 0,05 Gew.-% ≤ [Cu] ≤ 0,3 Gew.-%; 0,05 Gew.-% ≤ [Ga] ≤ 0,35 Gew.-%; 0,02 Gew.-% ≤ [Al] ≤ 0,3 Gew.-%.

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FIG 1

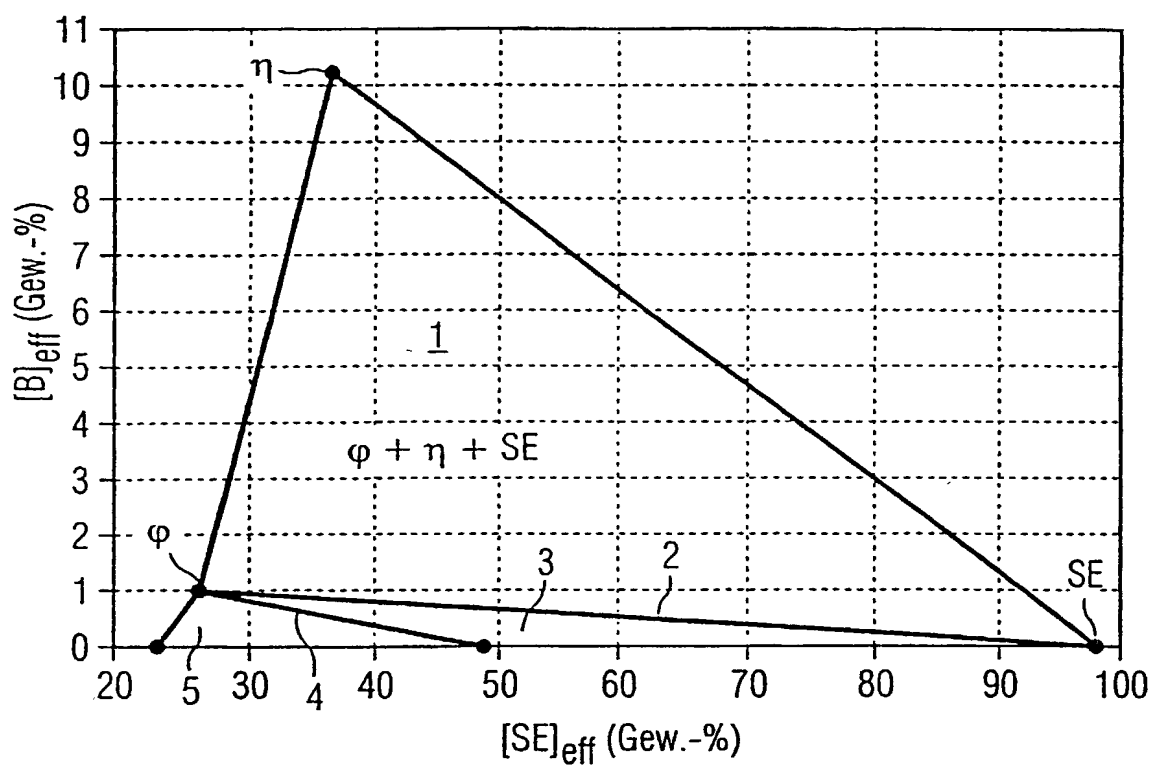


FIG 2

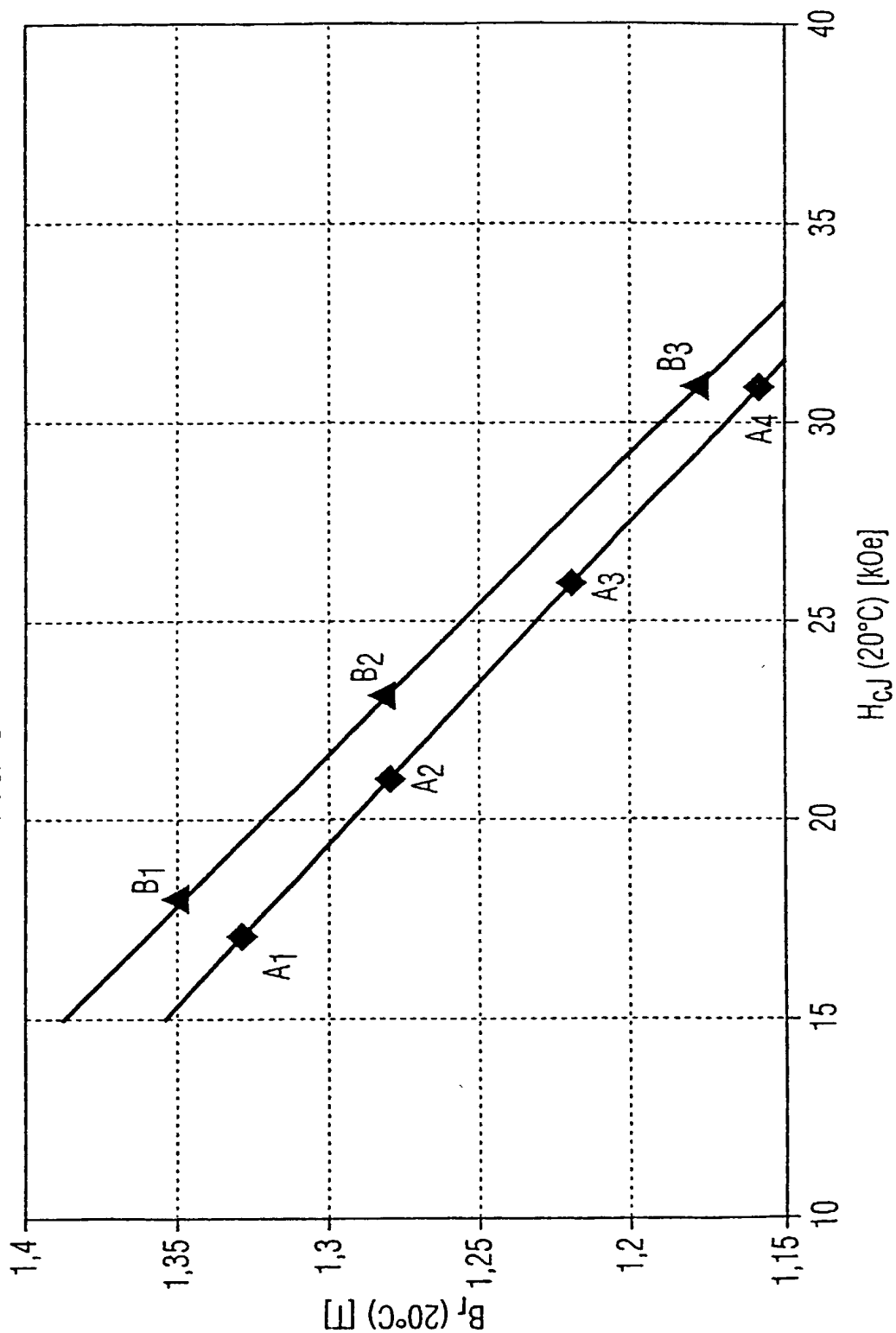


FIG 3

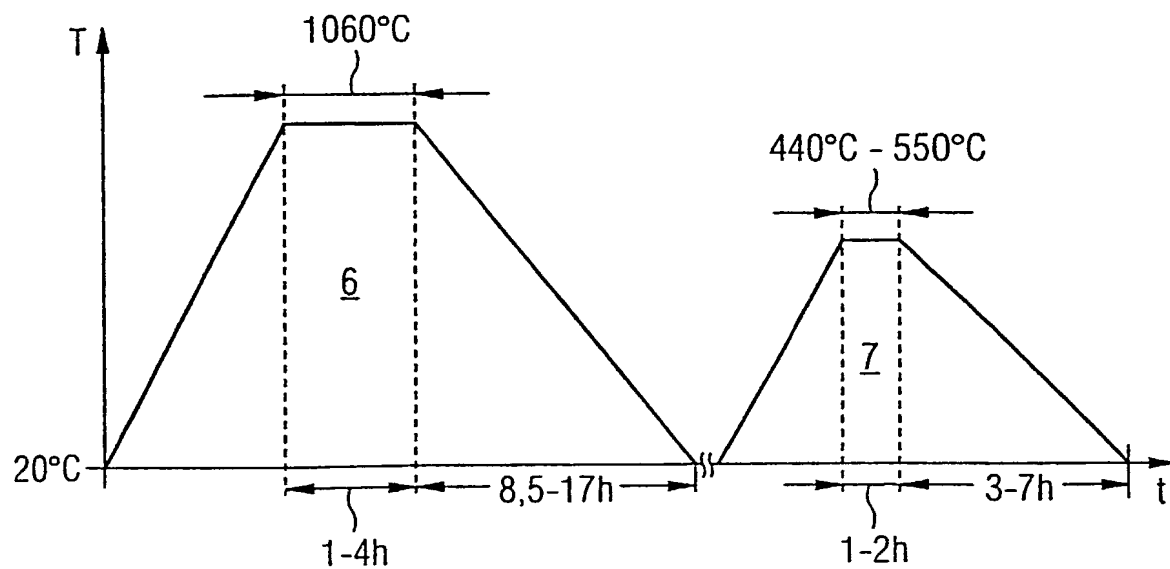
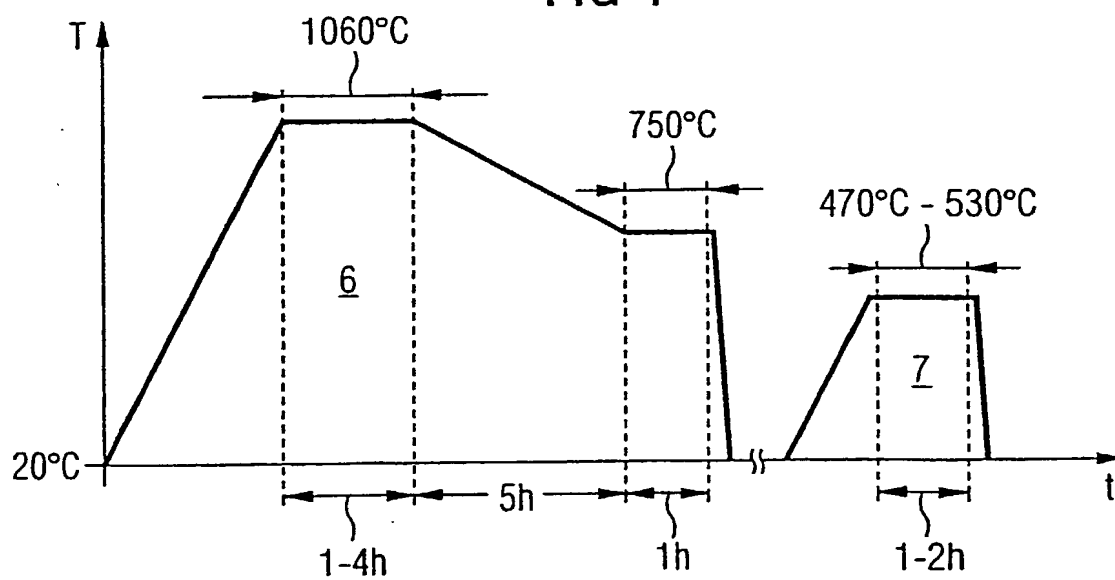
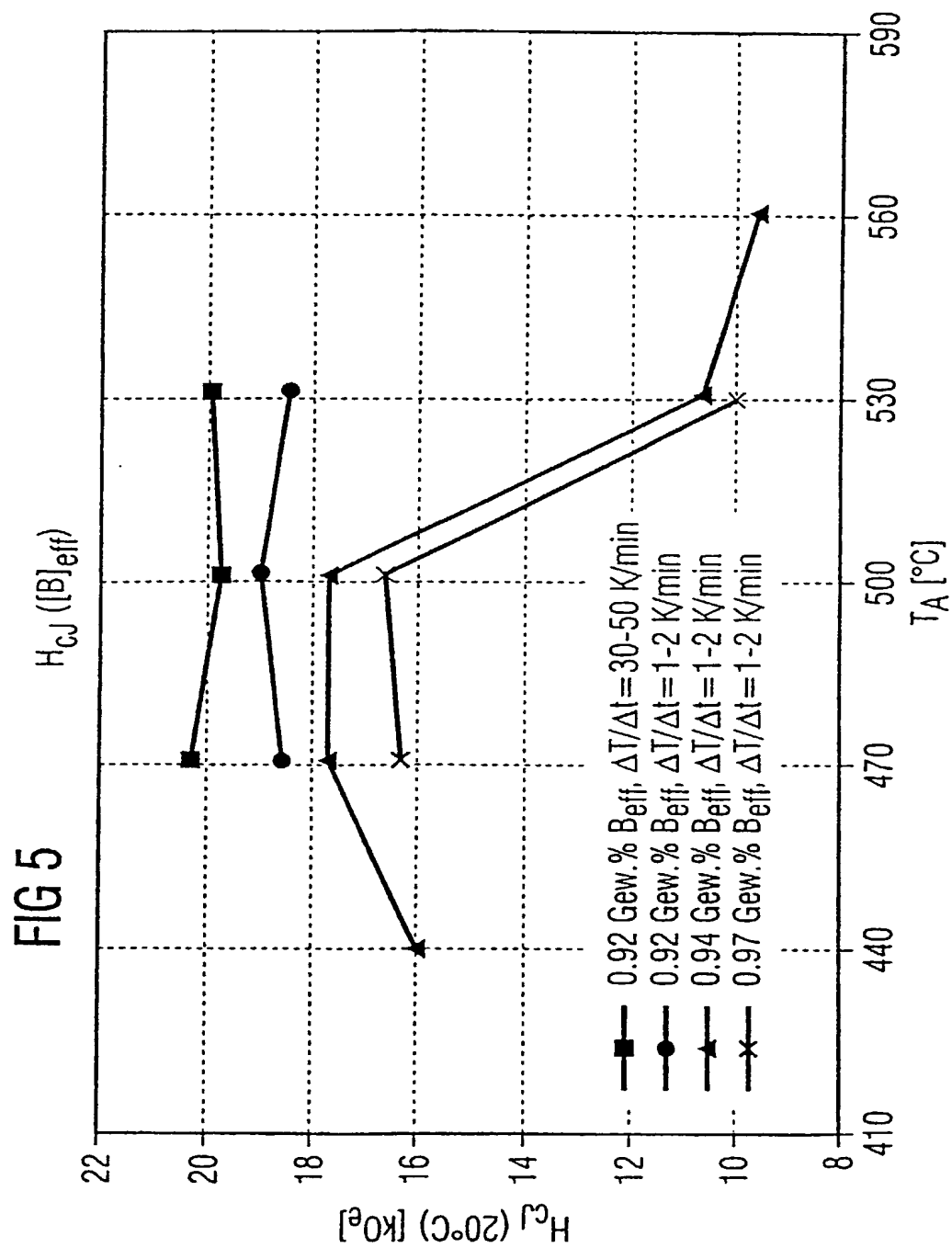


FIG 4





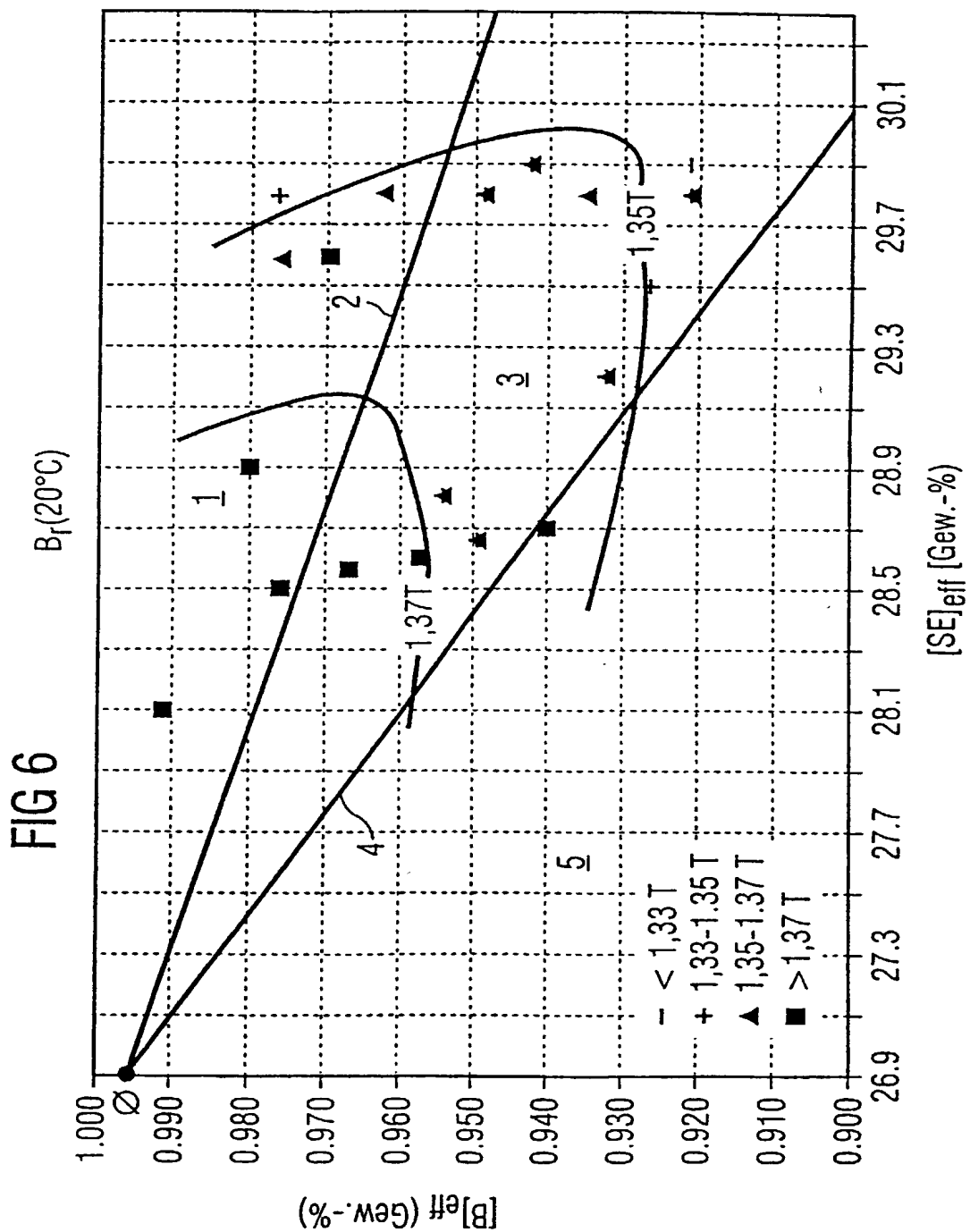


FIG 7

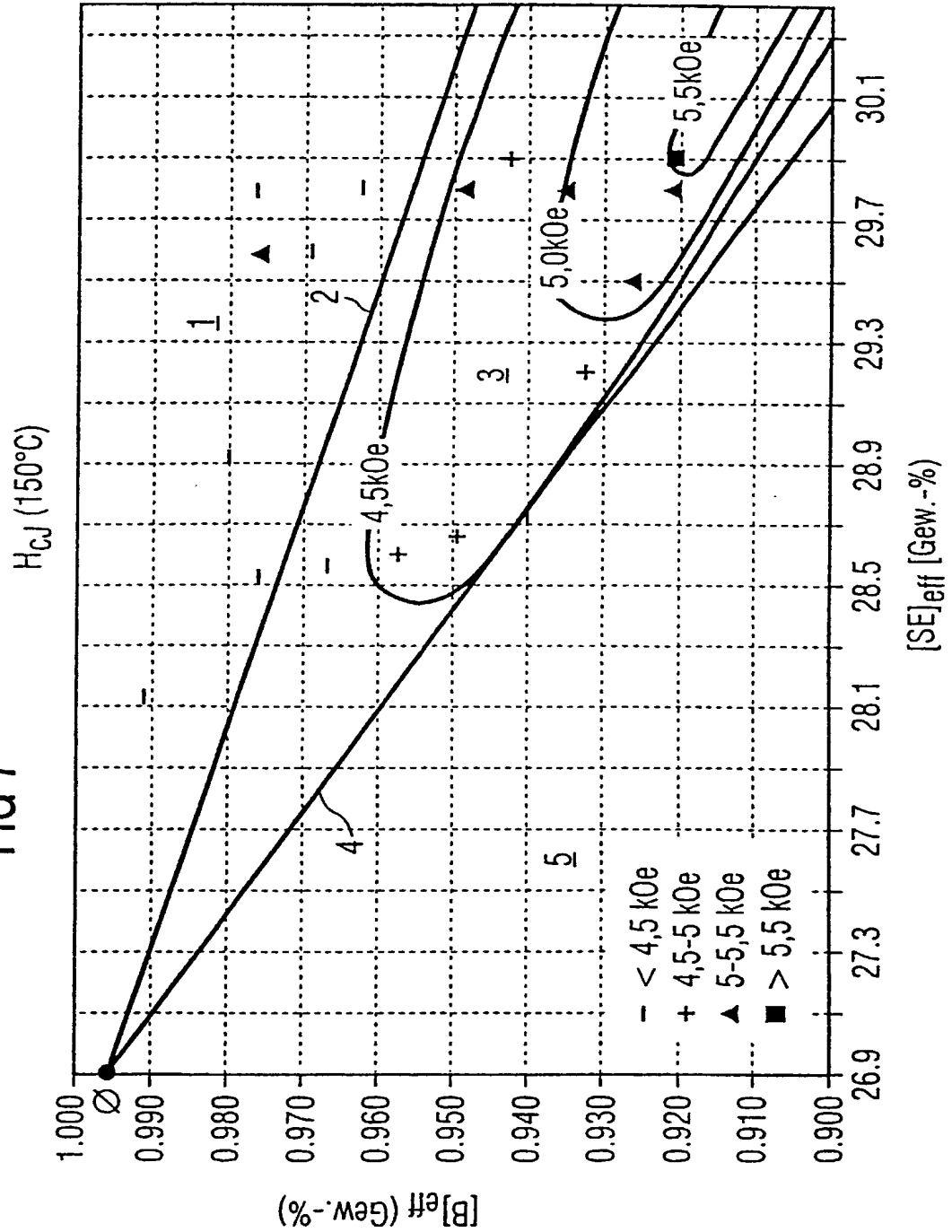


FIG 8

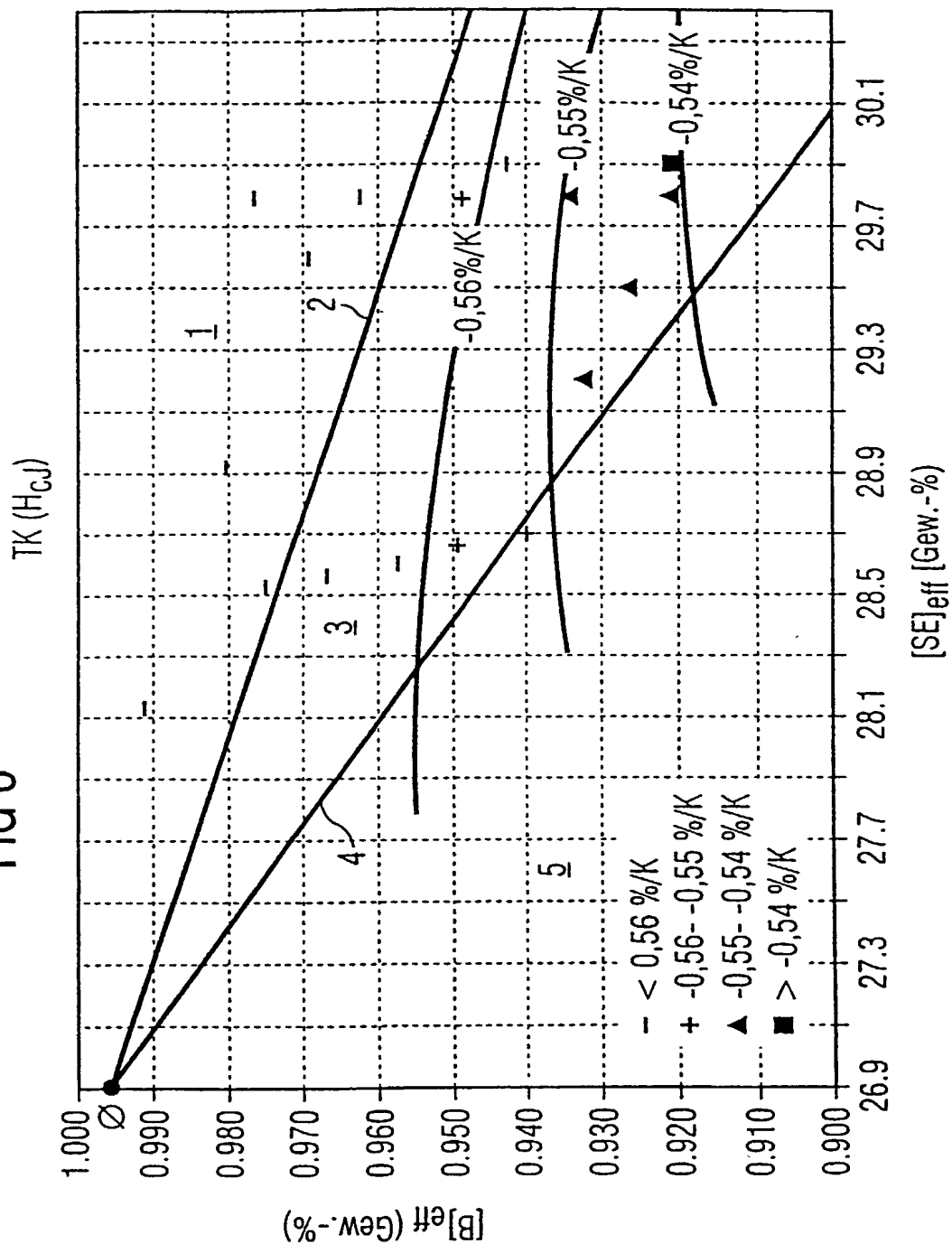


FIG 9

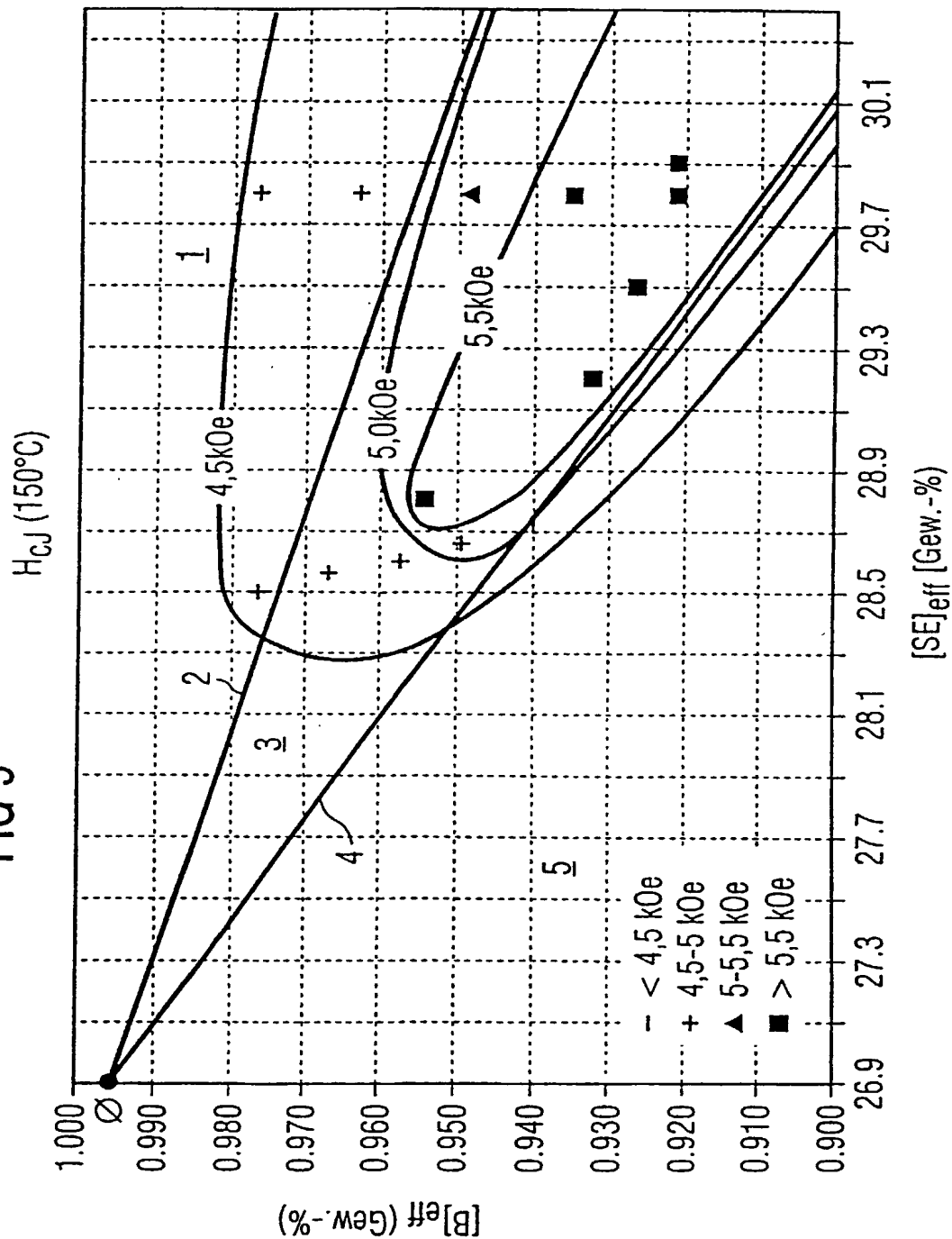
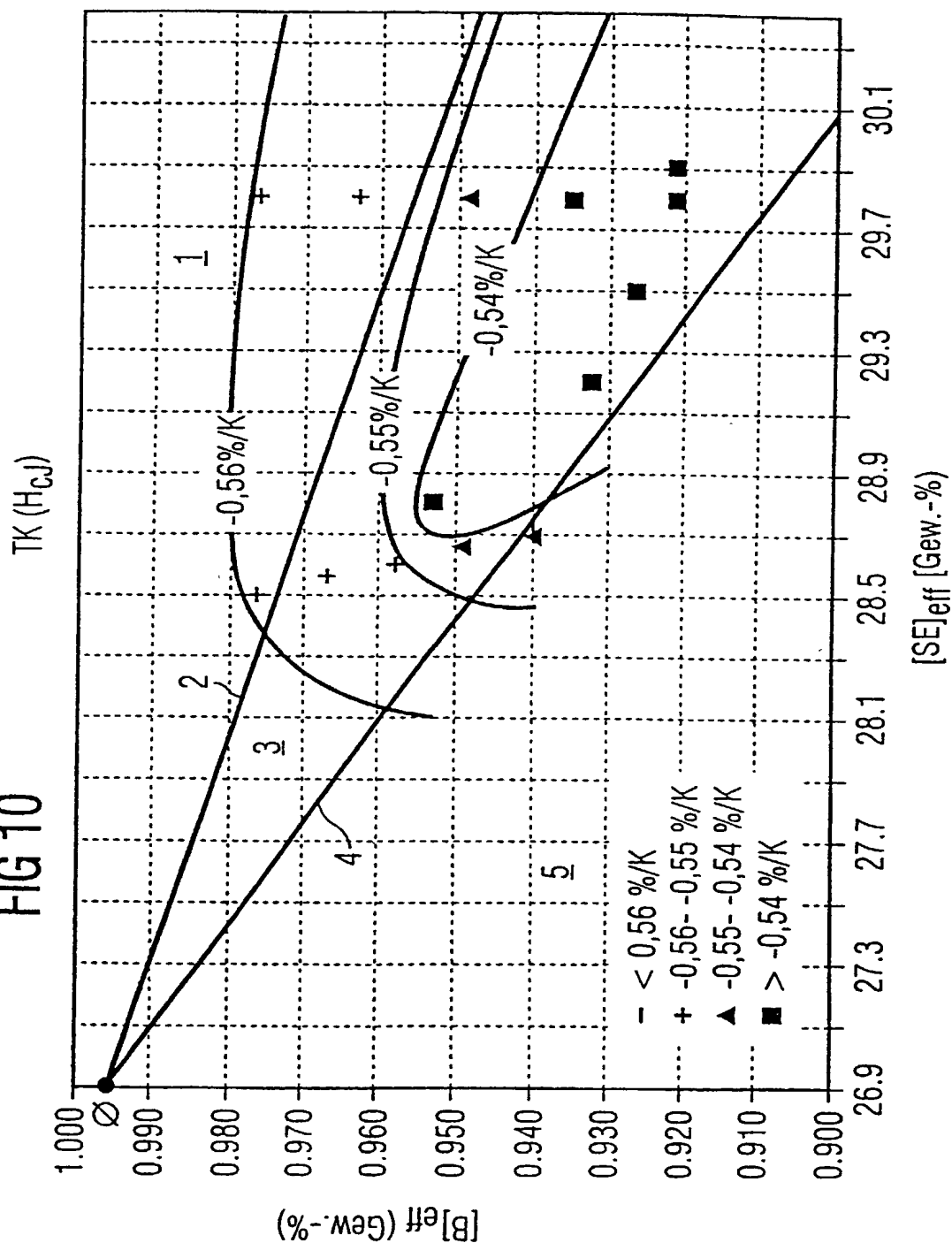


FIG 10



DECLARATION FOR PATENT APPLICATION

☒ Original

☐ Supplemental

☐ Substitute

☐ PCT

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below), or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Boron-Low Nd-Fe-B Alloy and Method for Producing Permanent Magnets on the Basis of Said Alloy

(Title of the Invention)

the specification of which (check one)

☐ is attached hereto

☒ was filed on 18 September 2000 as International Application No. PCT/EP00/09128
and was amended

(if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a) - (d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified, by checking the box below, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Applications			Priority Claimed		Copy Attached	
Application Number	Country	Foreign Filing Date (MM/DD/YYYY)	YES	NO	YES	NO
199 45 942.8	DE	09/24/99 (September 24, 1999)	X			x

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below and claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

Express Mail Label No. EL209599763US
U.S. National Phase of PCT/EP00/09128
Inventors: Matthias KATTER and Wilhelm FERNENGEL
Filed: 19 March 2002
Declaration for Patent Application
Filed:
Page 2

Parent Application Number	Filing Date	Status (Mark Appropriate Column Below)		
		Patented	Pending	Abandoned

As a named inventor, I hereby revoke all prior powers and appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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